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Formability of Aluminum Mild Detonating Fuse

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Abstract

Mild detonating fuse is an extruded aluminum tube that contains explosive material. Fuse prepared by a new supplier (Company B) exhibited a formability problem and was analyzed to determine the source of that formability problem. The formability problem was associated with cracking of the aluminum tube when it was bent around a small radius. Mild detonating fuse prepared by the existing supplier of product (Company A) did not exhibit a formability problem. The two fuses were prepared using different aluminum alloys. The microstructure and chemical composition of the two aluminum alloys were compared. It was found that the microstructure of the Company A aluminum exhibited clear signs of dynamic recrystallization while the Company B aluminum did not. Recrystallization results in the removal of dislocations associated with work hardening and will dramatically improve formability. Comparison of the chemical composition of the two aluminum alloys revealed that the Company A aluminum contained significantly lower levels of impurity elements (specifically Fe and Si) than the COMPANY B aluminum. It has been concluded that the formability problem exhibited by the COMPANY B material will be solved by using an aluminum alloy with low impurity content such as 1190-H18 or 1199-O.

Introduction

Mild detonating fuse (MDF) is a small diameter (0.041" nominal or 1.041mm) extruded aluminum tube containing explosive material. It is manufactured by filling an aluminum tube with explosive material and extruding the explosive containing tube using a conventional, multiple-die, cold, extrusion process. After extrusion this aluminum tube must be bent around a relatively small radius (0.070"). Traditionally this bend has not presented a problem, however, recently received lots of MDF exhibited cracking and fracture of the aluminum tube when bent. This report describes the source of this tube failure and recommends a solution. The solution is to use a higher purity aluminum alloy for the tube material, specifically an alloy that is extremely low in Fe and Si, like 1190-H18 or 1199-O.

Description of Problem

MDF extrusions have typically been supplied by (Company A). This material can be satisfactorily bent around small radiuses. Extrusions from a new supplier, (COMPANY B), are failing when bent around small radiuses even though both suppliers are using similar materials and extrusion processes.

The Company A Process

An aluminum tube is extruded through a series of progressively smaller extrusion dies until the final tube dimensions are reached. The material being extruded is a nominally pure aluminum alloy that was supplied with the following chemical analysis:

Table 1: Chemical Analysis of the Company A aluminum provided by Company A.

Element	Quantity
Al	99.99 weight %
В	22 ppm
Be	<10 ppm
Bi	<10 ppm
Cd	<10 ppm
Co	<10 ppm
Cr	<10 ppm
Cu	10 ppm
Fe	60 ppm
Ga	<10 ppm
Mn	<10 ppm
Ni	<20 ppm
Pb	<10 ppm
Sb	<20 ppm
Si	<20 ppm
Ti	<10 ppm
V	<10 ppm
Zn	<10 ppm
Zr	<10 ppm
Mg	20 ppm

This chemical analysis was performed using an inductively coupled plasma analysis technique (ICP). A number of elements are listed as being found in quantities less than 10 or 20 parts per million (PPM). These numbers likely represent the detection limit of the ICP analysis technique used. Thus, only Fe, B, Cu, and Mg are present in this material in detectable quantities. Despite these three impurities, this material is extremely pure aluminum, containing only 92 parts in 1 million (0.009%) that can be accounted for as something other than aluminum. Iron is the most common impurity element found in aluminum alloys because it is typically present in bauxite deposits and is difficult to completely remove from aluminum metal. It is the major impurity element in this material (60 PPM or ~ 0.006 wt%). Even at such a low level, iron in this alloy will precipitate upon solidification [1]. Boron is typically added to high purity aluminum alloys used for electrical wire. Boron scavenges transition metal elements like Ti, V, Cr,

and Zr thus increasing the electrical conductivity of the aluminum. Boron is the second major impurity element in this material (22 PPM or ~0.002 wt%). At this alloying level boron is either present in solid solution or is bound to Ti, V, Cr, and Zr atoms as complex intermetallics [2]. Like iron, copper is also a common impurity in aluminum alloys. It is the third most common impurity element in this material (10 PPM or ~0.001wt%). At this level copper is most likely in solid solution in the aluminum alloy [1].

The COMPANY B Process

An aluminum tube is formed by passing it through an initial swaging operation and then extruding it through a series of progressively smaller extrusion dies until the final tube dimensions are reached. The material being extruded is a commercially pure aluminum material (Alcoa Alloy 1090/1285) that was supplied with the following chemical analysis:

Table 2: Chemical Analysis of the COMPANY B material supplied by COMPANY B.

Element	Quantity (wt%)
Al	99.93
Si	0.0267
Fe	0.033
Cu	0.00
Mn	0.001
Mg	0.00
Zn	0.00
Ti	0.011
V	0.00
Ga	0.00
Pb+Cd	0.0000

The analysis technique used to assay this material is not specified. Like the ICP test, a number of elements are listed at levels of 0.00wt%. This likely indicates that they were not present in sufficient quantity to be detected by the analysis process. In this

material Fe, Si, Ti, and Mn are the major impurity elements. All four elements are common impurity elements in aluminum alloys and are within the specification limits for 1090 aluminum. They are present in significantly greater quantities than in the Company A material. Iron is present in this alloy at a level of 0.033wt% or ~ 330 PPM. This is a factor of 5 times more iron than in the Company A material. It will be present in the alloy as iron containing precipitates. Silicon is present at 0.0267wt% or ~260 PPM more than 13 times the maximum detectable silicon in the Company A material. At room temperature most if not all of this silicon is expected to precipitate in the aluminum alloy [1]. Ti is present at the 0.01wt% or ~ 100 PPM level and Mn is present at the 0.001 wt% or 10 PPM level. At these concentration both of these elements will remain in solid solution in the aluminum alloy [1]. While this aluminum alloy is a very pure material, it does contain significantly more impurity elements than the Company A material.

There is a difference in the extrusion procedure used by the two suppliers, but not a significant difference in the percent cold work received by each material. Percent cold work, or reduction in area, is calculated as follows:

$$\%CW = \left[\frac{A_0 - A_f}{A_0}\right] \times 100 \tag{1}$$

Where:

%CW = percent cold work

 A_{θ} = initial cross sectional area

 A_f = final cross sectional area

This equation is not rigorously correct for the reduction of a hollow tube. However, since the ratio of the outer diameter to the inner diameter for both tubes is approximately the same, it can be used to compare the two processes. If the starting and final diameters of each tube are put into equation (1) it is found that the Company A material received 98.91% cold work and the COMPANY B material received 99.87% cold work. This difference in cold work is not significant. Neither is the initial swaging operation received by the COMPANY B material. Any effects of this swaging operation on the microstructure of the COMPANY B material will be removed by the subsequent extrusion operations.

Alloy chemistry is the most significant difference in these two products, specifically the Fe and Si content. The Company A material has very little Fe and Si while the COMPANY B material contains significant quantities of both elements. Iron and silicon are important because they both precipitate in aluminum at room temperature. Precipitates in aluminum often form at grain boundaries and will limit grain boundary motion, thus preventing recrystallization.

Analysis of Samples

Samples of extruded MDF manufactured by each supplier were obtained. The explosive material contained in these samples was removed by chemical leaching before the samples were examined.

Metallography

All samples were mounted in epoxy and polished using standard metallographic techniques. They were then etched to reveal the grain structure of the aluminum. Figure 1 shows a longitudinal section of the COMPANY B material. One side of the extruded tube can be seen in this image. The outside of the tube is visible at the top of the image,

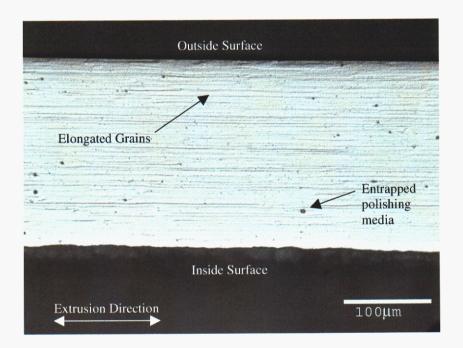


Figure 1: Longitudinal section of an COMPANY B extrusion. All of the grains in this sample are elongated in the extrusion direction. The small "inclusions" are most likely embedded polishing media.

the interior surface (the rougher surface) is visible near the bottom of the image. A large number of small, highly elongated grains can be seen in this material. The grains in this sample are elongated in the direction of the tube axis (the extrusion direction). This is consistent with a heavily cold worked extrusion. A number of small round objects can also be seen distributed throughout this image. These objects are most likely polishing

media that has become entrapped in the aluminum. It is unlikely that these objects were present in the aluminum during the extrusion process because they do not show elongation or evidence of fracture. Any inclusions present in the aluminum during the extrusion process should either be elongated in the extrusion direction or be heavily fractured.

Figure 2 shows a corresponding section of the extruded Company A material.

Again the outside of the tube can be seen at the top of the image and the inside surface

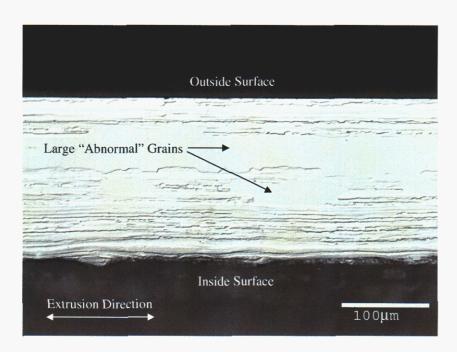


Figure 2: Longitudinal section of the Company A extrusion. Elongated grains are visible in this sample, however there are also a number of large grains typical of abnormal grain growth.

can be seen at the bottom of the image. This sample is noticeably different from the COMPANY B material. Most importantly it contains a few very large grains. It also contains a number of small elongated grains typical of a heavily extruded material. This

grain structure would be considered unusual in any material. It is especially unusual in a material that has been heavily extruded. Electron backscattered diffraction (EBSD) analysis was done in order to confirm that the grain structure seen in the metallographic samples is real.

Electron Backscattered Diffraction (EBSD) Analysis

EBSD analysis is an automated technique capable of mapping crystal orientations

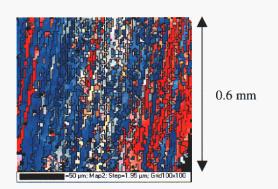


Figure 3. EBSD image of the COMPANY B material showing elongated grains.

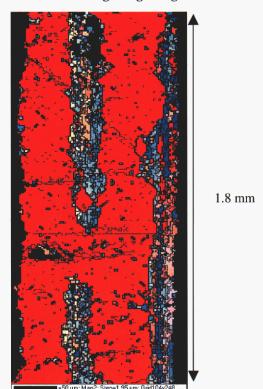


Figure 4. EBSD image of the Company A material showing its unusual grain structure.

in metallographically polished samples.

Automated crystal orientation mapping was performed using a JEOL 5900LV scanning electron microscope equipped with a

ThermoNoran ORKID electron backscattered diffraction (EBSD) system for orientation mapping. Orientation maps were collected by scanning the electron beam pixel-by-pixel across the area of interest. At each pixel, an EBSD pattern was collected, and automatically indexed and the orientation calculated. The orientations were displayed using colors to represent different orientations [3].

Figure 3 shows an EBSD map of the COMPANY B material. As expected, it

shows that this sample contains a large number of small elongated grains. Its fiber texture is largely <111> (blue grains) with some <100> fiber texture (red grains). This is consistent with extruded aluminum that has seen little or no subsequent annealing.

Figure 4 is an EBSD map of the Company A material. It shows that the small grains in this material are typically equiaxed and that what appear to be large grains are in fact extremely large single grains. The small grain regions have a mixed <111>, <100> fiber texture similar to the COMPANY B material. This suggests that the sample has not received any annealing after the extrusion process. The large grains must then be the result of dynamic recrystallization.

Dynamic recrystallization occurs in an alloy when new grains nucleate and grow while the material is being deformed. When this happens the material essentially self anneals at the deformation temperature. Work hardening typically associated with the deformation process does not occur because the recrystallizing grains remove dislocation pileups as soon as they form.

The extremely large grains in this material suggest that recrystallization continued for some time after deformation stopped. A few grains continued to grow and have formed large single crystal grains that extend for millimeters along the axis of the extruded tube. This phenomenon (one grain growing at the expense of almost all others) is known as abnormal grain growth.

The two self annealing phenomenon exhibited by the Company A material, dynamic recrystallization and abnormal grain growth, will result in a very low dislocation density in the final extrusion. Pure aluminum with a low dislocation density is very soft

and easily formed. This likely accounts for the better formability observed in the Company A material.

Inductively Coupled Plasma Mass Spectroscopy (ICPMS)

Recrystallization temperatures in aluminum vary considerably with alloy impurity content. In order to accurately compare the chemical compositions of the two materials, samples of each were analyzed using ICPMS analysis at Sandia National Laboratories. The Company A MDF (Inert), Lot#6593, non-hydrocompacted, Parent TU 19604, nominally 99.99% aluminum and Company B MDF (Inert) Piece R-14, non-hydrocompacted, Parent TU 50020, nominally 99.9% aluminum are shown below. The samples were prepped to determine trace metals content, in triplicate by dissolving ~0.04g of material in 8ml of 37.5% HCl. They were then diluted to 100mls. Metals that were present at levels greater than 5ppm were identified and measured. Values are the average and standard deviation of the three measurements for the Company B material and two measurements for the Company A material. Aluminum content was calculated by difference.

Table 3: ICPMS Analysis of the Company A material performed at Sandia National Laboratories.

Element	Concentration
Si	30.3±2.8 ppm
В	11.7±1.0 ppm
Ga	11.4±0.3 ppm
Zn	9.3±0.7 ppm
Mn	7.5±0.7 ppm

Mo	6.1±0.2 ppm
Cu	5.1±1.1 ppm
Al	99.99%

Table 4: ICPMS analysis of the COMPANY B material performed at Sandia National Laboratories.

Element	Concentration
Fe	316±25 ppm
Si	174±7 ppm
Ti	111±1 ppm
V	43.0±5.0 ppm
Ga	42.1±3.1 ppm
Zn	37.4±3.7 ppm
Cu	14.6±2.7 ppm
Mn	9.5±0.2 ppm
Zr	7.3±0.3 ppm
Al	99.92%

This chemical analysis confirms the differences in Fe and Si content that were noted in the manufacturers analysis. The Company A material has significantly less Fe and Si, than the COMPANY B material. Also, the overall impurity content of the Company A material is significantly lower than the overall impurity content of the COMPANY B material.

It is well known that impurities in aluminum can affect recrystallization temperature [4, 5]. Iron and silicon are known to strongly affect recrystallization temperature. As the amount of these elements in the aluminum alloy increases, the

recrystallization temperature increases [4]. The lower Fe and Si content of the Company A material is likely responsible for its recrystallization behavior.

It is important to note that the Company A material may not be recrystallizing at room temperature. In fact, the presence of some small grains in the sample suggest that it is not changing at room temperature. According to the manufacturer's documents a post-extrusion anneal is not performed on this material. It is also reasonable to assume that it was not heated at any time after extrusion, since heating could cause unwanted changes in the explosive material contained inside the extruded tubes. There may have been a small amount of heating of the aluminum due to adiabatic heating and friction during the extrusion process. That this heating could have caused the Company A aluminum to recrystallize. Adiabatic heating (or heating due to deformation) is a function of deformation rate. It is conceivable that the Company A and COMPANY B materials were deformed at different rates and that this resulted in a difference in adiabatic heating.

Summary, Conclusions, and Recommendations

Two samples of mild detonating fuse (MDF) aluminum extrusions were examined to determine why one sample (COMPANY B) could not be formed acceptably when the other sample (Company A) could. The manufacturers specifications show that both materials received similar amounts of cold work during extrusion. They also showed that there was a significant difference in composition between the two materials. The formable material (Company A) contained significantly less Fe and Si than the unformable material (COMPANY B).

Metallographic analysis of the unformable material (COMPANY B) showed that its microstructure consisted of small elongated grains typical of a heavily extruded material. The microstructure of the formable material (Company A) consisted of small equiaxed grains and very large abnormal grains. This microstructure is consistent with a material that underwent dynamic recrystallization during the extrusion process. Further chemical analysis of both materials confirmed that the Company A material contained significantly less Fe and Si than the COMPANY B material.

The lower alloy content of the Company A material resulted in a reduced recrystallization temperature. It is likely that heating during the extrusion process caused this material to recrystallize. The recrystallized material is significantly softer and more formable than the COMPANY B material which did not recrystallize after annealing.

In order to ensure that future MDF extrusions will be formable, an aluminum alloy containing very little Fe and Si should be selected. Aluminum 1190-H18 and 1199-O are the purest commercially available aluminum alloys on today's market [6]. These materials have significantly less alloy content (especially Fe and Si) than the alloy used by COMPANY B. However, they do contain more alloy content than the material used by Company A.

If extrusions made from 1190-H18 and 1199-O do not exhibit acceptable formability two options can be considered. One is a moderate temperature post-extrusion anneal. Annealing the extrusion even for a short time will increase its formability. The risk associated with heating an explosive material must be carefully considered if this option is pursued. The second option is to change the extrusion schedule so that the

aluminum receives less cold work. This will involve starting the process with aluminum tube that is as close to the final diameter and length as possible.

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References

- 1. Hansen, Constitution of Binary Alloys. Second ed. 1958: McGraw Hill.
- Hall, A. and J. Economy, The Al(L) + AlB12 -> AlB2 Peritectic Transformation and Its Role in the Formation of High Aspect Ratio AlB2 Flakes. Journal of Phase Equilibria, 2000. 21(1): p. 63-69.
- 3. Schwartz, A.J., K. M, and B.L. Adams, eds. *Electron Backscatter Diffraction in Materials Science*. . 2000, Kluwer Academic/Plenum Publishers: New York.
- 4. Humphreys, F.J. and M. Hatherly, *Chapter 6: Recrystallization of Single-Phase Alloys*, in *Recrystallization and Related Annealing Phenomena*. 1995.
- Marshall, G.J., R.A. Ricks, and P.K.F. Limbach, Controlling Lower Temperature Recovery and Recrystallization in Commercial Purity Aluminum. Materials Science And Technology, 1991: p. 263-269.
- 6. Lampman, S. and S. Henry, ASM Handbook: Volume 2 Properties and Selection: Nonferrous Alloys and Special-Purpose Materials, ed. S. Lampman. Vol. 2. 1990: ASM International.

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